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Chemical Analysis of Core Soil Samples - Upper Ottawa Street Landfill Study

Reference Paper 3

REFERENCE 3

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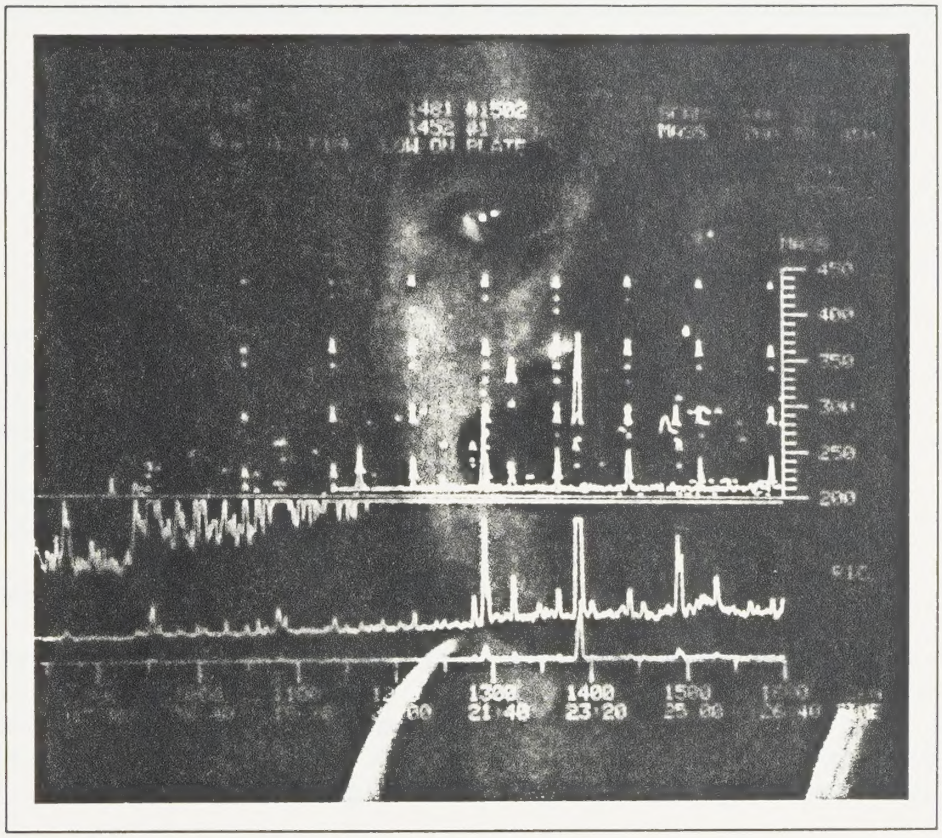

CHEMICAL ANALYSIS

OF CORE SOIL SAMPLES

FROM BH #2 AND BH #4

MANN TESTING LABORATORIES LTD.

PROFESSIONAL
ANALYTICAL SERVICES

MANN TESTING LABORATORIES LTD.
80 GALAXY BLVD., UNIT 10, REXDALE, ONTARIO M9W 4Y8
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October 1, 1982

Anne Koven
Upper Ottawa Street
Landfill Site Study
42 James Street, S.
Suite 33
Hamilton, Ontario
L8P 2Y4

Dear Anne:

This is the final report for the analysis of core soil samples taken from Bore Holes #2 and #4. This report contains information gathered on the base neutral and acid extracts of the soils, PCB and chlorinated organics of the soils, and voltile fraction headspace analysis of the soils.

In summarizing the results, the extracts contained large amounts of hydrocarbons and polynuclear aromatic hydrocarbons in the soils. The polychlorinated biphenyls (PCB's) were found in highest concentration in Bore Hole #4. The headspace analysis detected chlorinated solvents, specifically the chlorinated ethylenes, in the soil samples.

If you have any questions regarding this analyses, please do not hesitate to contact me.

Yours truly,

Stan Hazan

SH/rc

Members: Association of Official Racing Chemists, American Chemical Society, Canadian Society of Forensic Science, Chemical Institute of Canada, American Society for Testing and Materials, Canadian Federation of Independent Business. Better Business Bureau, Association of the Chemical Profession of Ontario, Association of Professional Engineers of the Province of Ontario, Canadian Association of Fire Chiefs.

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Chemical Analyis of Core Soil Samples From BH #2 and BH #4

Six core samples obtained from bore holes #2 and #4 were analyzed for organic compounds, specifically chlorinated organics and polyaromatic hydrocarbons. The samples were selected on the basis of odour by Dr. Bournes and Anne Koven of the Upper Ottawa Street, Landfill Committee. The six soil samples selected were taken at different depths from the two bore holes and are listed below:

Soil #1	Bore Hole #2	25 foot depth
Soil #2	Bore Hole #2	45 foot depth
Soil #3	Bore Hole #2	55 foot depth
Soil #4	Bore Hole #2	65 foot depth
Soil #5	Bore Hole #4	55 foot depth
Soil #6	Bore Hole #4	80 foot depth

The soil samples were contaminated with high concentrations of waste oil which could mask the presence of toxic compounds in low concentrations. In order to enhance the detection of toxic compounds present in low concentrations, a florosil cleanup procedure was instituted to remove the non-polar oil/PAH fraction, thus allowing lower detection limits on the slightly more polar compounds. The acid extracts were derivatized with diazomethane in order to chromatograph the very polar compounds.

Base-Neutral Extraction Procedure

A portion of the soil sample was weighed wet then dry to determine the % water content. The wet soil was extracted although the amount of soil reported here is in terms of dry weight.

Soil #1	20G
Soil #2	10G
Soil #3	12G
Soil #4	17G
Soil #5	12G
Soil #6	17G

The soil samples were all spiked with 300 micrograms deuterated anthracene (C12D10), and extracted three times with 90:10 dichloromethane:isopropanol. The organic fraction was filtered and washed with alkaline water (pH 12). The organic fraction was passed thru a Na2SO4 filter and rotoevaporated without heat to a volume of 5 millilitres. The extract was further concentrated under a stream of nitrogen. The extract was picked up with 50:50 ether:hexane and placed on top of the pre-conditioned sodium sulfate/florosil column. The column was eluted with 250 ml of hexane (cut 1), then 250 ml of 6% diethyl ether in hexane (cut 3), 200 ml of 50% diethyl ether (cut 5). Each cut was rotoevaporated and concentrated to one millilitre. Preliminary tests on cut 4 and cut 5 indicated very little contamination present in these fractions. Therefore, analysis of cuts 4 and 5 was not performed. Two of 1000

microlitres was injected onto a 20M DB-5 capillary column and analyzed by the Finnigan 4500 GC/MS/DS.

Acid Extraction Procedure

Approximately 5 grams of each soil sample was washed three times with 6N NaOH solution (pH 12). The water was filtered into a separatory funnel and extracted twice with 100 ml of dichloromethane. The dichloromethane was discarded, the aqueous fractions were made acidic (pH 2) and extracted twice with 100 ml of dichloromethane. The organic fractions were rotoevaporated to near dryness, then transfered to vials containing diazomethane (in excess). The vials were warmed for one hour and allowed to react overnight. The extracts were evaporated to dryness then reconstituted to 1.0 ml of which 2 ul was injected onto the GC/MS.

Results

The soil was generally alkaline (pH > 9) and reacted violently upon addition of acid (HCl). The reaction resulted in heat being produced, violent bubbling and foaming, and emmission of H2S-like gas.

The base-neutral extracts were divided into three florosil cuts, each cut containing characteristic components. In the case of the first florosil fraction, the major components were aliphatic hydrocarbons, (octane, nonane, decane, undecane, etc.) aromatic hydrocarbons (benzene, toluene, xylenes, etc.), and poly nuclear aromatic hydrocarbons (naphthalene, anthracene, pyrene, etc.). The second florosil cut contained mainly phthalate esters and the third cut contained phthalates and high molecular weight phenols. The acid extracts contained large amounts of chain acids and diacids (nonanoic acid, nonanedioic acid as the methyl esters), phenols, and benzene alkyl acids (benzoic acid, benzene acetic acid, benzene propanoic acid etc.) and phthalic acids as the methyl esters. Each soil sample is summarized as the compounds detected and confirmed by GC/MS. As requested, non-toxic compounds such as chain hydrocarbons, chain acids etc, although in high concentrations, are not reported. The chromatograms and raw library search results are found in Appendix 1. Many of these library search results are erroneous due to low purityor poor match.

Compounds Detected in Soil Sample #1

Soil #1 BN Extract

FLOROSIL CUT #1 MS File #1887

- hydrocarbons (waste oil)
- polynuclear aromatic hydrocarbons (PAH)
- sulfur (SO2 and/or S8)

FLOROSIL CUT #2 MS File #1936

- phthalates
- phenols

FLOROSIL CUT #3 MS File #2051

- phthalate esters
- phenols
- phenanthrene carboxylic acids/methyl esters

ACID EXTRACT & DIAZOMETHANE MS File #2036

alkyl benzoic acids

- methoxy benzoic acids
- phenols
- dimethyl trisulfide
- methyl thiopropene
- phthalate esters

Compounds Detected in Soil Sample #2

Soil #2 BN Extract

FLOROSIL CUT #1 — MS File #1888

- polynuclear aromatic hydrocarbons
- hydrocarbons (waste oil)
- phthalate esters

FLOROSIL CUT #2 — MS File #1939

- cyclohexylamino-benzothiazole
- phenols
- phthalates
- trimethylcyclohexene-1-methanol

FLOROSIL CUT #3 — MS File #2052

- phthalates

ACID EXTRACT & DIAZOMETHANE — MS File #2039

- aklyl benzene acids
- alkyl benzene dioic acids
- phenols
- phenoxyl benzoic acid
- methoxy[(methoxy phenyl)methyl]benzene
- dimethoxy benzoic acids
- methoxy phenyl ethanone
- methoxy phenyl propanone
- hydroxy benzene propanoic acid
- phthalates

Compounds Detected in Soil Sample #3

Soil #3 BN Extract

FLOROSIL CUT #1 — MS File #1889

- hydrocarbons (waste oil)
- polynuclear aromatic hydrocarbons (PAH)
- phthalates

FLOROSIL CUT #2 — MS File #1941

- phthalate ester
- phenol

FLOROSIL CUT #3 — MS File #2053

- alkyl ketone ($C_{14}H_{20}O_2$)
- phthalate esters
- octyl diphenyl phosphate

ACID EXTRACT & DIAZOMETHANE — MS File #2047

- phenanthrene carboxylic acid derivatives
- 4,4'-methylene-bis-phenol
- methoxy [(methoxy phenyl)methyl]benzene

Compounds Detected in Soil Sample #4

Soil #4 BN Extract

FLOROSIL CUT #1 — MS File #1890

- hydrocarbons (waste oil)
- polynuclear aromatic hydrocarbons (PAH)
- phthalates
- sulfur (SO2 and/orS8)

FLOROSIL CUT #2 — MS File #1942

- phthalate esters

FLOROSIL CUT #3 — MS File #2054

- phthalates
- tributyl phosphate

ACID EXTRACT & DIAZOMETHANE — MS File #2048

- alkyl benzoic acids
- methoxy and dimethoxy benzoic acids
- methoxy phenyl acetone
- methoxy[(methoxy phenyl)methyl]-benzene
- phthalates

Compounds Detected in Soil Sample #5

Soil #5 BN Extract

FLOROSIL CUT #1 — MS File #1867

- alkyl benzenes
- polynuclear aromatic hydrocarbons (PAH)
- alkyl hydrocarbons (waste oil)

FLOROSIL CUT #2 — MS File #1870

- phenols
- phthalates

FLOROSIL CUT #3 — MS File #1871

- phenols
- phthalates

ACID EXTRACT & DIAZOMETHANE — MS File #2050

- alkyl benzoic acids
- methoxy benzoic acids
- methoxy benzaldehyde
- phenoxy benzoic acids
- phenanthrene carboxylic acids
- methoxy[(methoxy phenyl)methyl]-benzene
- methoxy-biphenyl
- methoxy benzisothiazole
- phthalates

Compounds Detected in Soil Sample #6

Soil #6 BN Extract

FLOROSIL CUT #1 — MS File #1891

- alkyl benzenes
- polynuclear aromatic hydrocarbons (PAH)
- phthalates
- alkyl hydrocarbons (waste oil)

FLORISIL CUT #2 — MS File #1943

- phenols
- phthalates

FLOROSIL CUT #3 — MS File #2055

- phenols
- phthalates
- tributylphosphate
- C9-ketone

ACID EXTRACT & DIAZOMETHANE — MS File #2049

- alkyl benzoic acids
- methoxy benzoic acids
- methoxy benzoisothiazole
- methoxy biphenyl
- biphenyl-2-ol
- phthalates
- phenanthrene carboxylic acids
- methoxy[(methoxy phenyl)methyl]benzene

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TELEX:05-822787(LYNJON MTL)

TO: Mann Testing Laboratories Ltd.
80 Galaxy Blvd., Unit 10
Rexdale, Ontario
M9W 4Y8

Attention: Mr. John Martin.

DATE: September 9, 1982.

CLIENT ORDER #: -

REPORT #: NL-594

RE: Analysis of Sediments for PCB.

Sir,

Six (6) sediment samples were analysed for PCB by Environment Canada methods. Results (dry weight basis) are shown in Table I. The limit of detection was 0.01µg/g.

TABLE I - Concentration of PCB in Sediment(µg/g)

Sample	Aroclor 1242	Aroclor 1254	Aroclor 1260
BH#2, 25ft	0.38	0.32	0.21
BH#2, 45ft	0.45	0.30	0.21
BH#2, 55ft	0.86	0.54	0.41
BH#2, 65ft	0.48	0.38	0.25
BH#4, 55ft	0.92	1.05	0.61
BH#4, 79½ft	5.62	4.03	1.55

No significant amount of non-PCB chlorinated organics was noted in any sample.

Yours sincerely,

NOVALAB LIMITED

J.D. Fenwick, Ph.D., P.Chem.

JDF:sm

All reports are the confidential property of clients. Publication of statements, conclusions or extracts regarding our reports is not permitted without our written approval. Any liability attached thereto is limited to the fee charged. Unless otherwise arranged samples will be discarded 90 days after receipt.

Headspace Analysis for Volatile Components in Soil

The frozen soil samples were thawed and approximately 10 grams placed into a 40 ml vial with a teflon septum. The vial was warmed up in an oven to 90 degrees celsius. A gas-tight syringe was pumped several times in the pressurized headspace, and a 10 ml volume of gas was injected directly onto the concentrator, equipped with a 50-metre Superox capillary column. The eluants were analyzed by mass spectrometry. The analysis is by no means quantitative, however all parameters remaining equal, one can relate between the individual soil samples. The raw library search results and chromatograms can be found in Appendix 2. Som of the identifications are poor due to poor fiit and low purity. The actual compounds detected and confirmed are listed on the following pages.

(1) Bore Hole #2, 25' Depth MS File #1925

This sample was found to be relatively clean of contaminants. Instrument and laboratory background were higher than the actual contaminants.

(2) Bore Hole #2, 45' Depth MS File #1946

This sample contained alkyl hydrocarbons, aromatic hydrocarbons, and low levels of chlorinated solvents. Overall, the contaminants were in low concentration.

Compounds Detected

- aliphatic H/C
- benzene
- toluene
- xylenes
- tetrachloroethylene

(3) Bore Hole #2, 55' Depth MS File #1947

This sample was found to be much more heavily contaiminated than the preceeding two samples. The soil appeared to be nearly saturated with aliphatic and aromatic hydrocarbons. Chlorinated solvents were also detected.

Compounds Detected

- C2-benzenes
- C3-benzenes
- C4-benzenes
- C5-benzenes
- C3-C6 ketones
- C3-C8-alcohols
- tetrahydrofuran
- naphthalene
- dichloroethane
- dichloroethylene
- trichloroethylene
- tetrachloroethylene

(4) Bore Hole #2, 65' Depth MS File #1948

The concentration of toluene and most other contaminants is approximately twice that of the preceeding sample. The chlorinated solvents were found in slightly lower concentration than the preceeding sample.

Compounds Detected

- C3-C12 hydrocarbons
- benzene
- C1-benzene (toluene)
- C2-benzene
- C3-benzene
- C4-benzene
- C5-benzene
- C3-C8 ketones
- C3-C8 alcohols
- tetrahydrofuran
- naphthalene
- dichloroethane
- dichloroethylene
- trichloroethylene
- tetrachloroethylene

(5) Bore Hole #4, 55' Depth MS File #1875

Forty ml of headspace was used here instead of the 10 ml used in the other soil samples. This sample contained approximately double the chlorinated solvents concentration of the preceeding sample #4, whereas the toluene concentration was lower.

Compound Detected

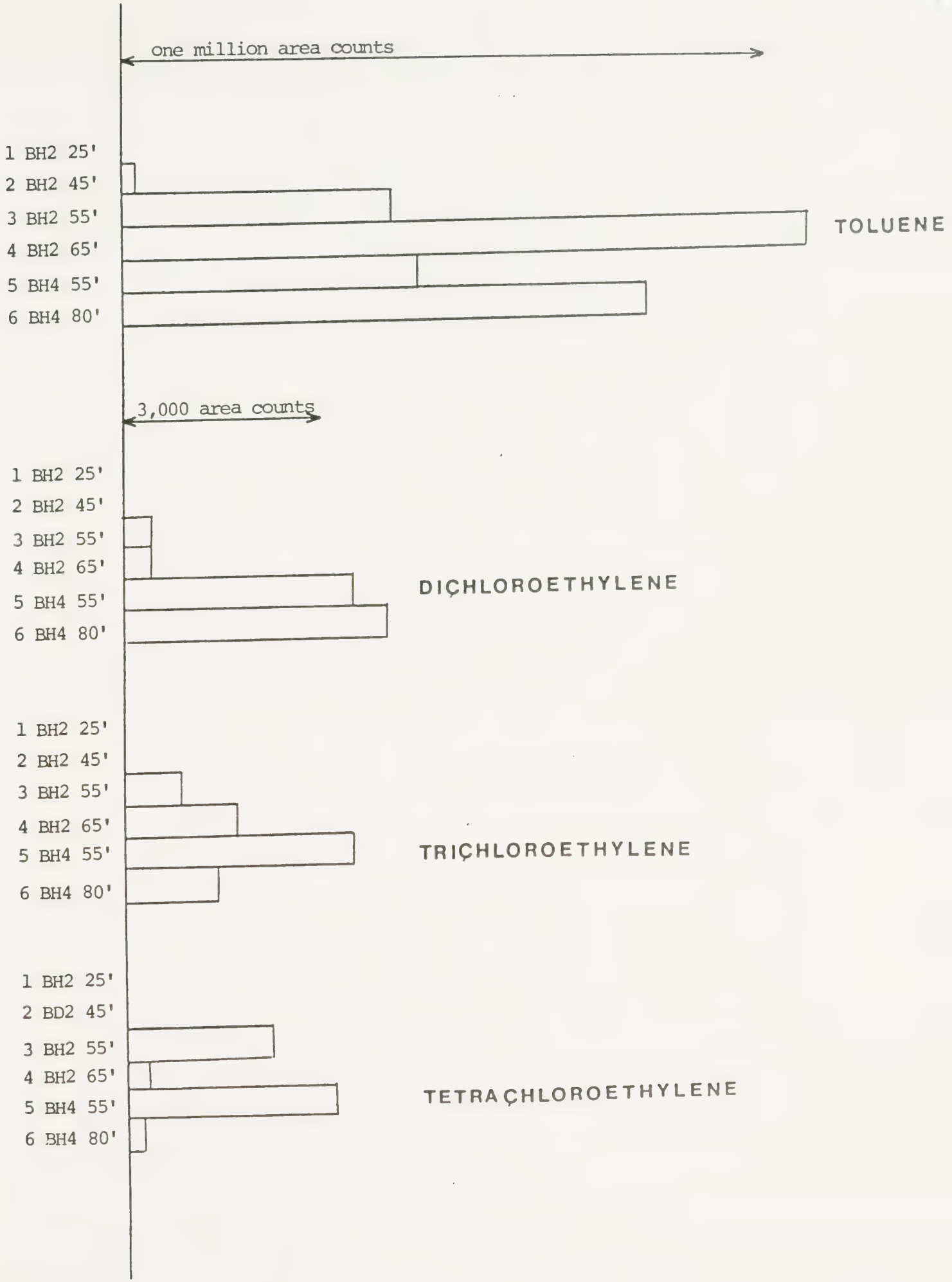
- C3-C12 hydrocarbons
- benzene
- C1-benzene (toluene)
- C2-benzenes
- C3-benzenes
- C4-benzenes
- C5-benzenes
- C3-C8 ketones
- C3-C8 alcohols
- dichloroethylene
- trichloroethylene
- tetrahydrofuran
- naphthalene

(6) Bore Hole #4, 80' Depth MS File #1949

This sample contained more toluene and chlorinated solvents that sample #5.

Compounds Detected

- C3-C12 hydrocarbons
- benzene
- C1-benzene (toluene)
- C2-benzene
- C3-benzene
- C4-benzene
- C5-benzene
- dichloromethane
- dichloroethylene
- trichloroethylene
- tetrachloroethylene
- C3-C8 ketones
- C3-C8 alcohols
- naphthalene


one million area counts
1 BH2 25'
2 BH2 45'
3 BH2 55'
4 BH2 65'
5 BH4 55'
6 BH4 80'
TOLUENE
3,000 area counts
1 BH2 25'
2 BH2 45'
3 BH2 55'
4 BH2 65'
5 BH4 55'
6 BH4 80'
DICHLOROETHYLENE
1 BH2 25'
2 BH2 45'
3 BH2 55'
4 BH2 65'
5 BH4 55'
6 BH4 80'
TRICHLOROETHYLENE
1 BH2 25'
2 BD2 45'
3 BH2 55'
4 BH2 65'
5 BH4 55'
6 BH4 80'
TETRACHLOROETHYLENE

Summary

The major reason for fractionating the base neutral extracts was to remove the high oil/PAH interference so as to allow lower detection limits for any compounds of high toxicity. The final results for these soils indicate that there are no heavy chlorinated organics in any of the soils in high concentrations. This was confirmed by Novalab Ltd. that performed the PCB analysis. The PCB analysis is carried out using an EC detector which produces good selective responses for chlorinated species. The samples contained high concentrations of oil and PAH as expected.

The headspace analysis showed the presence of some chlorinated solvents present in the soil samples. The six samples also contained high amounts of alkyl benzenes.

The chromatograms and mass spectra for each analysis is on file at the lab (disc and hard copy) and can be produced upon request.

The compounds reported in each soil samaple are those in which we have a high degree of confidence as to identity.

We recommend that the chlorinated solvents, PAH's, alkyl benzenes, and phenols should form part of the list of compounds to be quantified in the leachate samples. Since there exists so many isomers of PAH's and alkyl benzenes, it may be advantageous to simply quantify selected representative compounds of each class. This approach would speed up and simplify the quantitation process, and yield equally significant results.

Approximate Concentrations of PAH's in Soil Samples*

Concentration in PPM

MW	Compound Name	#1	#2	#3	#4	#5	#6
128	Napthalene	5.6	1.0	0.4	0.3	32.8	9.5
142	C1-naphthalene/and isomers	0.8	0.2	0.1	0.1	4.2	3.9
156	C2-naphthalene/and isomers	0.4	0.3	0.1	0.2	2.9	3.7
170	C3-naphthalene/and isomers	0.2	0.2	0.1	0.1	0.9	2.8
184	C4-naphthalene/and isomers	0.3	0.2	0.1	0	0.2	1.4
152	Biphenylene/acenaphthalene	0.7	0.2	0.1	0.2	1.0	0.3
154	1,2 dihydroacenaphthalene	0.8	0.4	0.2	0.4	2.0	0.7
168	Dibenzofuran	0.9	0.2	0.1	0.3	2.4	1.0
166	Phenalene/Fluorene	0.7	0.4	0.1	0	3.7	1.7
178	Phenanthrene	6.3	9.6	3.6	8.5	24.3	13.5
178	Anthracene	0.6	0.9	0.5	0.7	5.6	1.2
188	D10-anthracene (int. std.)	15.0	30.0	25.0	17.6	25.0	17.6
192	C1-anthracene/and isomers	0.4	0.5	0.2	0.3	2.5	1.4
206	C2-anthracene/and isomers	0.3	0.4	0.1	0.1	1.3	0.9
220	C3-anthracene/and isomers	0.2	0.2	0.1	0.1	0.8	0.3
202	Pyrene	4.5	6.3	2.5	6.2	23.5	9.0
202	Fluoranthene	3.0	5.0	2.2	4.0	22.5	7.8
216	C1-pyrene/and isomers	0.1	0.4	0.1	0.2	3.0	0.6
230	C2-pyrene/and iosmers	0.1	0.1	0	0	0.5	0.1
242	C3-pyrene/and isomers	0.1	0.1	0.1	0.1	1.4	0.2
228	Chrysene/or other isomers	0.7	1.6	0.7	1.1	9.5	2.9
252	Benzo[ ]pyrene/and isomers	0.6	2.0	1.1	1.5	7.3	2.4
276	Benzo[ ]perylene/and isomers	0.1	0.3	0.2	0.2	0.6	0.4

Approximate Concentrations

*based on D10-Anthracene - not corrected for relative response

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